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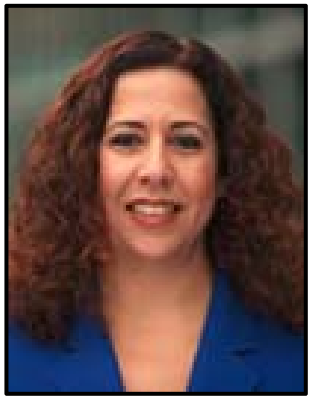
Advanced Nanocrystalline Alloys as Alternatives for Repair and Replacement of High Wear Parts

April 5, 2018

SERDP DOD • EPA • DOE | ESTCP

Welcome and Introductions



Rula A. Deeb, Ph.D.
Webinar Coordinator

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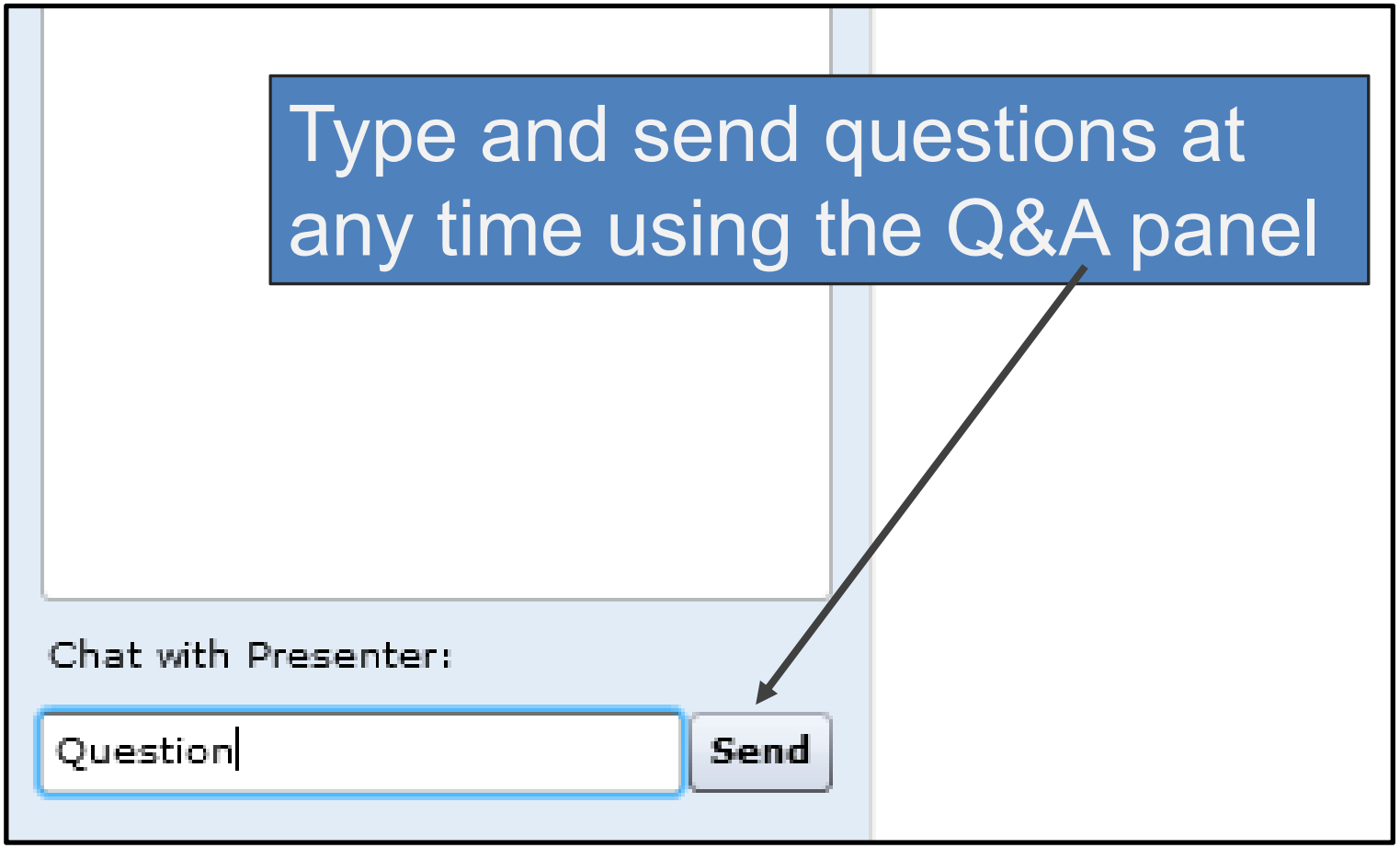
Webinar Agenda

- **Webinar Logistics** (5 minutes)
  Dr. Rula Deeb, Geosyntec Consultants
- **Overview of SERDP and ESTCP** (5 minutes)
  Dr. Robin Nissan, SERDP and ESTCP
- **Nano Cobalt Alloys and Composites as Alternatives for Chromium and Nickel Plating in Repair Operations**
  Dr. Jonathan McCrea, Integran Technologies (25 minutes + Q&A)
- **Qualification Testing of Electroformed Nanostructured Cobalt-Alloy Bushings** (25 minutes + Q&A)
  Dr. Alan Grieve, Naval Air Warfare Center Aircraft Division
- **Final Q&A session**

How to Ask Questions



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SERDP and ESTCP Overview

Robin Nissan, Ph.D.
SERDP and ESTCP

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SERDP

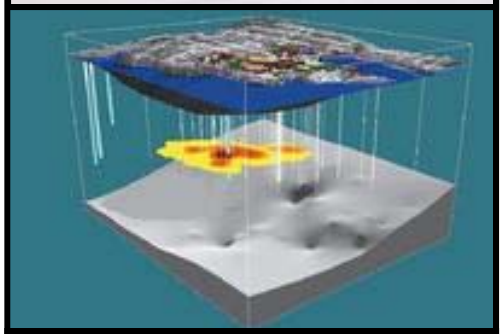
- Strategic Environmental Research and Development Program
- Established by Congress in FY 1991
  - DoD, DOE and EPA partnership
- SERDP is a requirements driven program which identifies high-priority environmental science and technology investment opportunities that address DoD requirements
  - Advanced technology development to address near term needs
  - Fundamental research to impact real world environmental management

ESTCP

- Environmental Security Technology Certification Program
- Demonstrate innovative cost-effective environmental and energy technologies
  - Capitalize on past investments
  - Transition technology out of the lab
- Promote implementation
  - Facilitate regulatory acceptance

Program Areas

1. Environmental Restoration
2. Installation Energy and Water
3. Munitions Response
4. Resource Conservation and Resiliency
5. Weapons Systems and Platforms



SERDP and ESTCP Webinar Series

Date	Topic
April 19, 2018	Sediment Volume Search Sonar
May 3, 2018	The Defense Coastal Estuarine Research Program (DCERP): Enhancing Ecosystem-Based Management at a Coastal Installation
May 17, 2018	Tools to Evaluate Passive and Active Remedial Approaches at Chlorinated Solvent Sites
May 31, 2018	Resonant Acoustic Mixing of Energetic Material Formulations
June 14, 2018	Approaches to Managing Threatened, Endangered and At-Risk Bird Species
June 28, 2018	Energy Sustainable Wastewater Treatment Systems for Forward Operating DoD Installations

For upcoming webinars, please visit

http://serdp-estcp.org/Tools-and-Training/Webinar-Series

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Save the Date

SERDP • ESTCP
SYMPOSIUM
2018 | Enhancing DoD's Mission Effectiveness

A three-day symposium showcasing the latest technologies that enhance DoD's mission through improved environmental and energy performance

November 27 - 29, 2018

Washington Hilton Hotel

Registration is coming soon

Nano Cobalt Alloys and Composites as Alternatives for Chromium and Nickel Plating in Repair Operations

Jonathan McCrea, Ph.D., P.Eng.
Integran Technologies Inc.

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Agenda

- Problem statement
- Proposed solution
- Project objectives
- Results to date
- Benefits to DoD
- Next steps

Problem Statement

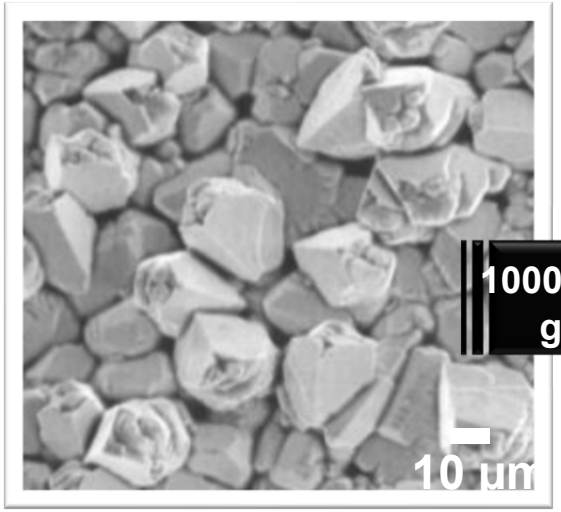
- Chromium (Cr) and nickel (Ni) plating in repair operations poses environmental and health risks
  - Ni compounds are carcinogens*
  - Cr(VI) is a carcinogen
    - OSHA PEL reduced from 52 to 5 $\mu g/m^3$
- Need an alternative material that is environmentally benign and cost-effective

*U.S. Department of Health and Human Services 13th Report on Carcinogens on October 2, 2014

Background: What is a Nanostructured Metal?

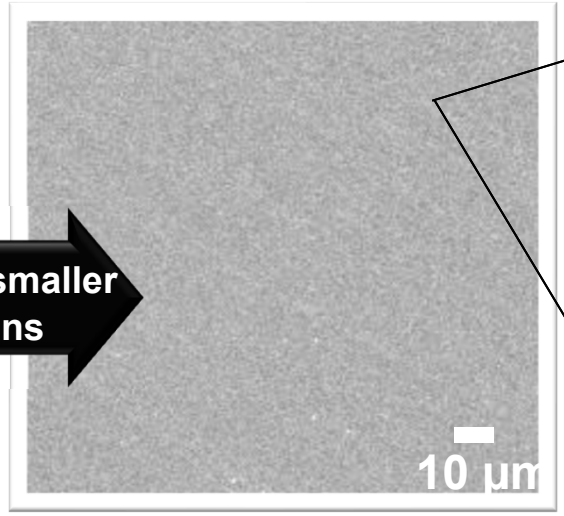
Average Grain Size 10 to 100 Nanometers

Conventional metals
Grain size = 10-100 µm

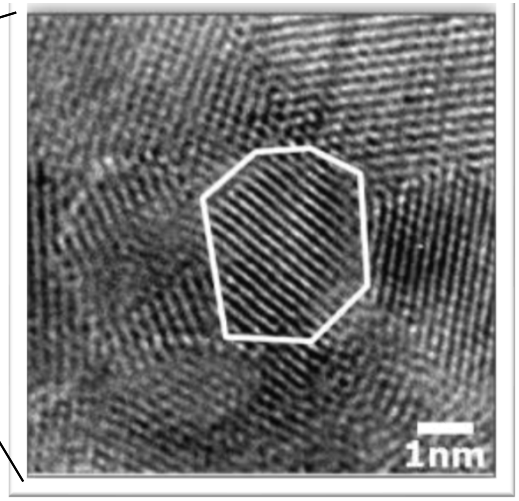


1000x smaller grains

Nanovate™ metals
Grain size = ≤ 20 nm



A grain of nanocrystalline material by HR-TEM

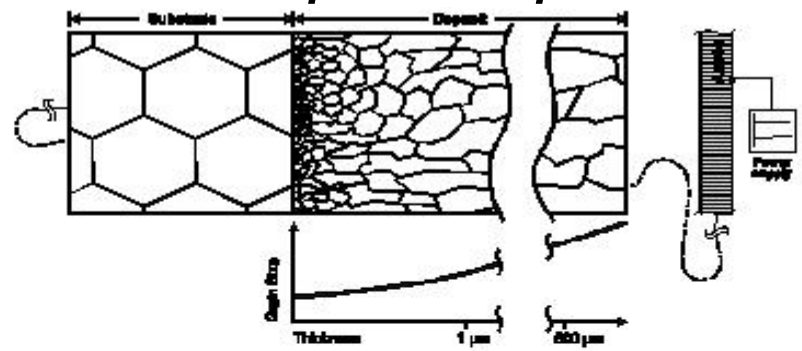


Decreasing Grain Size Dramatically Improves Hardness and Strength			
Property	**Units**	**Conventional Ni (20 µm)**	**Nanovate Ni (20 nm)**
Yield Strength	MPa	100	900
Ult. Tensile Strength	MPa	400	1400
Vickers Hardness	kg/mm^2	140	450

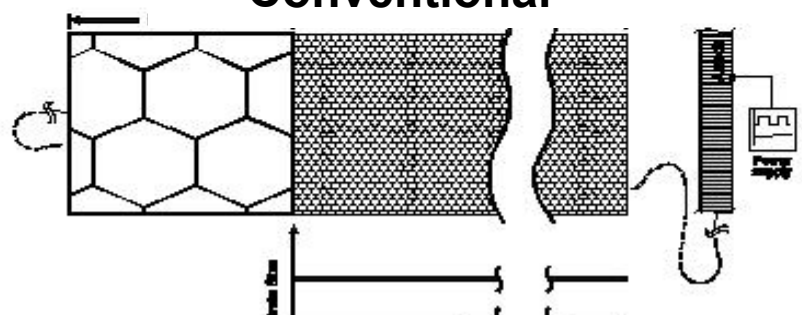
Nanovate Cobalt Phosphorus (nCoP)

Pulsed Electrodeposition Achieves Unique Properties

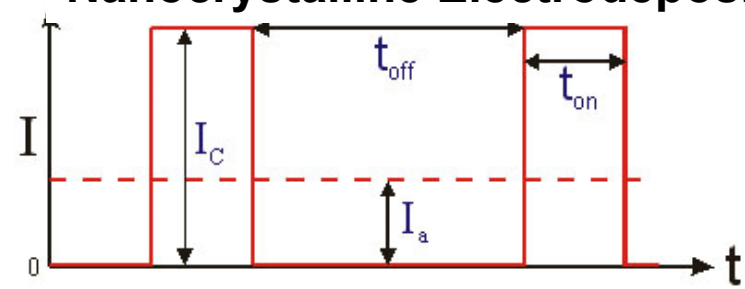
- Pulse plating favors nucleation of new grains over growth of existing grains
  - Ultra-fine grain structure throughout the entire thickness of the coating, right from the substrate interface
- Pulsed electrodeposition from aqueous solutions
  - fully dense metal with a nanocrystalline grain size deposited
- Can fine grained nanostructures be achieved without using pulse plating?



Conventional

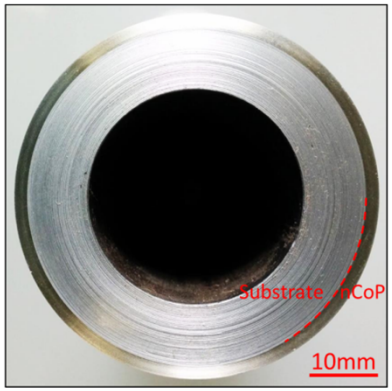


Nanocrystalline Electrodeposits



Solution: NanovateTM CoP R3010

- Electrodeposited nanocrystalline cobalt-phosphorus alloy
- Cost effective
- Significantly improved corrosion resistance, lower friction, ductility and toughness compared to engineering hard chrome (EHC)
- US military specification (MIL-DTL-32502)/Commercial specification, AMS 2428



Nanovate™ CoP

Process Comparison

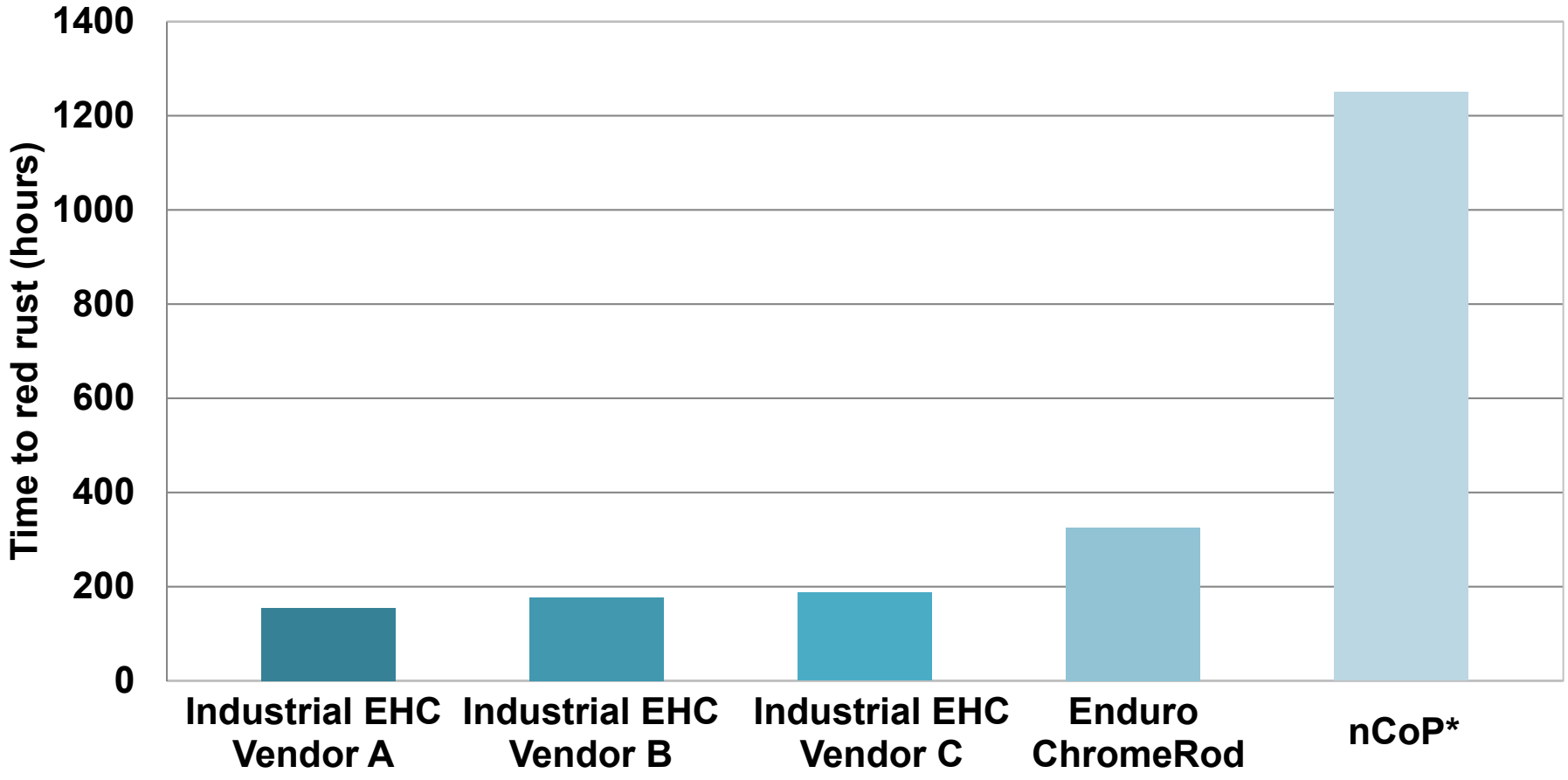
- At least 5X faster than chrome plating
- Increased throughput
- Can replace several hard chrome tanks
- Bath is stable

Parameter	Nano CoP	EHC
Electrodeposition method	Pulse / DC[1]	DC
Part geometries	LOS and NLOS	LOS and NLOS
Efficiency (%)	85 - 95	15 - 35
Deposition rate (inches/hour)	0.002 - 0.008	0.0005 - 0.001
Emission analysis	Below OSHA limits	Cr^{+6}
Bath temperature (°F)	185 / 167[1]	140

LOS = Line of sight; NLOS = Non-line of sight
[1]Currently in demonstration/ validation stage

Process Corrosion

nCoP vs. EHC, B117



*Test was stopped after 1200 hours with no corrosion present on Nanovate CoP samples

Corrosion

CoP vs. EHC, B117

Nanovate CoP

Pit, pore and crack-free

Hard Chrome

Microcracked

Nanocrystalline Plating

A Very Well Established Technology

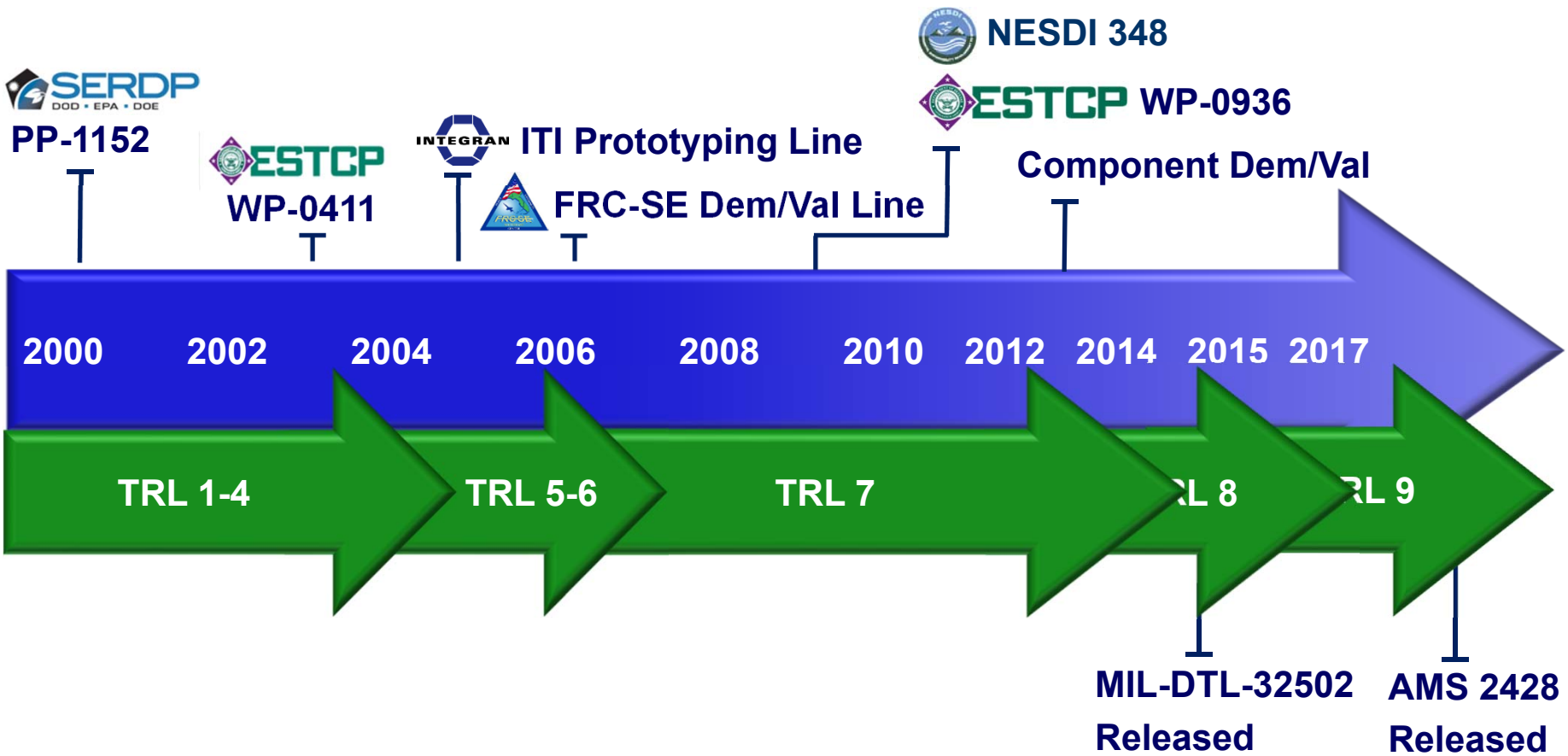
- Plating tanks in place at Integran and FRC-SE
- Demonstration/validation successfully completed in 2015

Process line at FRC-SE

650-gallon plating tank at Integran since 2005

250-gallon tank at FRC-SE

Technology Maturity (TRL)



Currently being used for production OEM parts for a number of defense and industrial hydraulic components/applications

SERDP WP-2609

Project Objective

Replace Cr and Ni Plating in Repair Operations

- Develop and validate an alternative repair technology based on electrodeposited nanostructured cobalt alloy
- Must meet or exceed the properties and performance of:
  - Electroplated nickel coatings
    - Standards: MIL-STD-868A and AMS-QQ-N-290
  - Electrolytic hard chrome coatings
    - Standard: MS-QQ-C-320
  - Selective plating, repair, and rebuilds
    - Standard: MIL-STD-865C

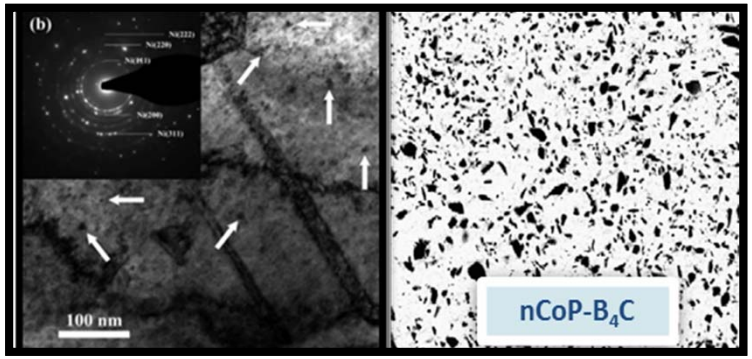
Project Team

- Integran Technologies Inc.
  - Specialists in electrodeposition and properties of nanostructured materials (Jonathan McCrea, Ijya Srivastava, Gino Palumbo)
- Cirrus Materials Science
  - Specialists in in-situ formation of nanocomposites in plating chemistries (Glen Slater, Chris Goode, See-Leng Tay)
- Corrdesa
  - Expertise in modeling of electrochemical corrosion potentials (Alan Rose, Siva Palani)
- NAVAIR FRCSE
  - DoD stakeholder, end user, input for requirements (Ruben Prado, John Benfer)

Project Objectives

Nanovate CoP Process and Performance Enhancement

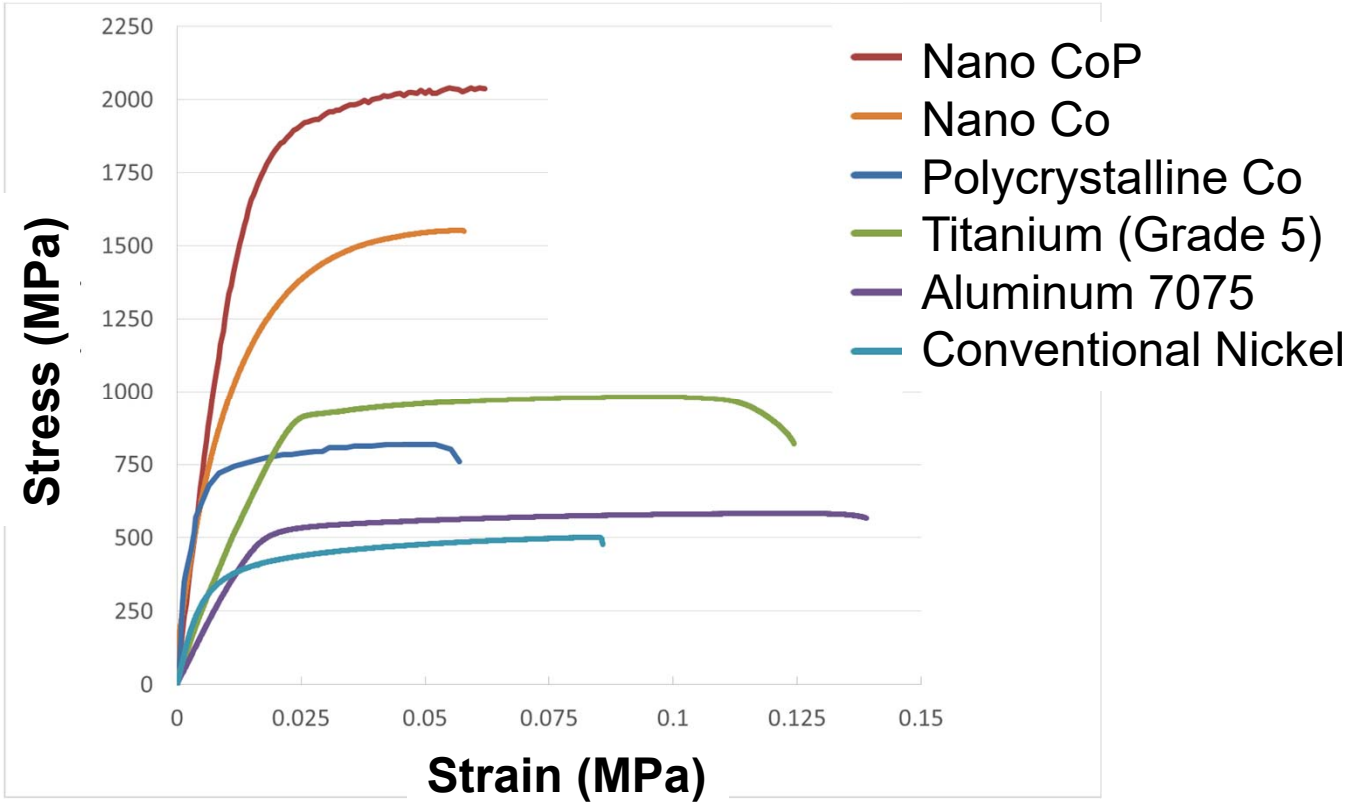
1. CoP process optimization
   - Dem/Val of direct current plating
   - Dem/Val of lower plating temperature
   - Plating on aluminum
2. Investigate CoP metal matrix composites for enhanced wear properties
   - Co-deposition of suspended particles
   - Novel in-situ particle formation and deposition of nanoparticles (Cirrus dopant technology)
3. Brush plating for repair operations
   - Evaluation and optimization of equipment to enable brush plating for specialized repair for CoP and CoP-composite systems



Stress-Strain Comparison

nCo and nCoP Alloys vs. Conventional Materials

- High strength and toughness of nCo alloys make them well suited for repairing structural components
- Coating can facilitate a structural repair without flaking or spalling during high levels of strain



Objective 1: CoP Process Optimization

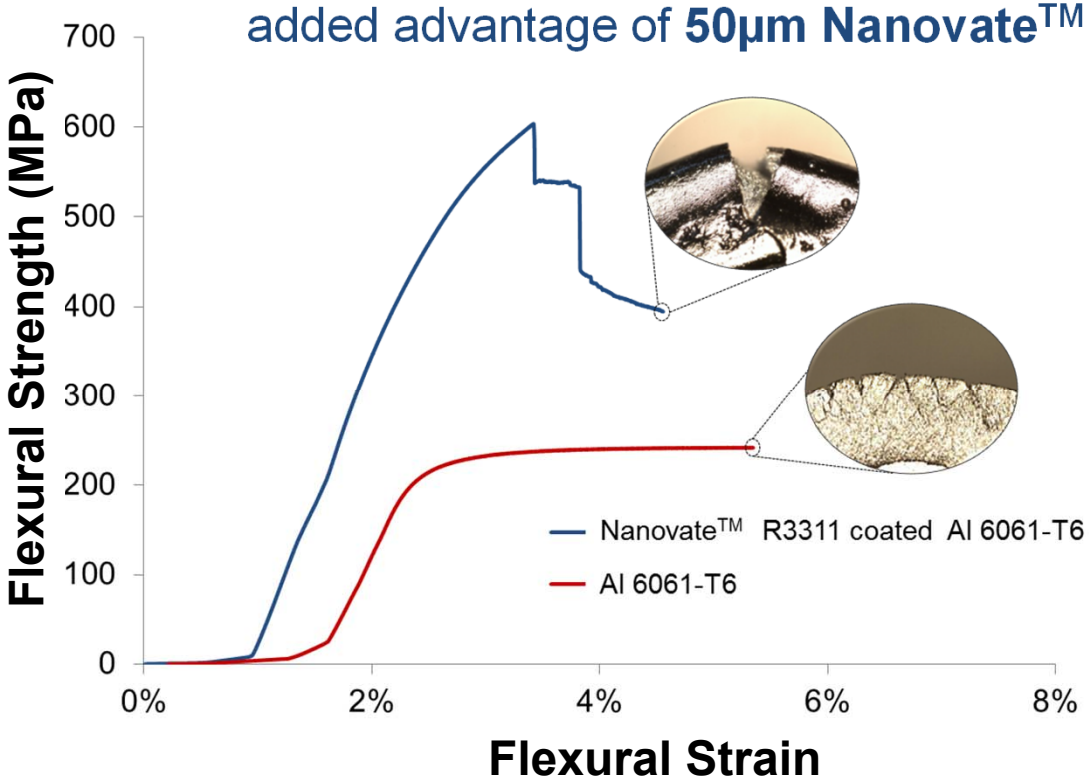
- **Objective**: Optimize CoP process for repair operations based on conventional DC rectifiers and reduce operating temperatures
- **Status**: Low-temperature, DC-compatible CoP plating developed and validated
  - Additional benefit: operating window for current density increased
  - No major change to key deposit properties such as hardness and wear

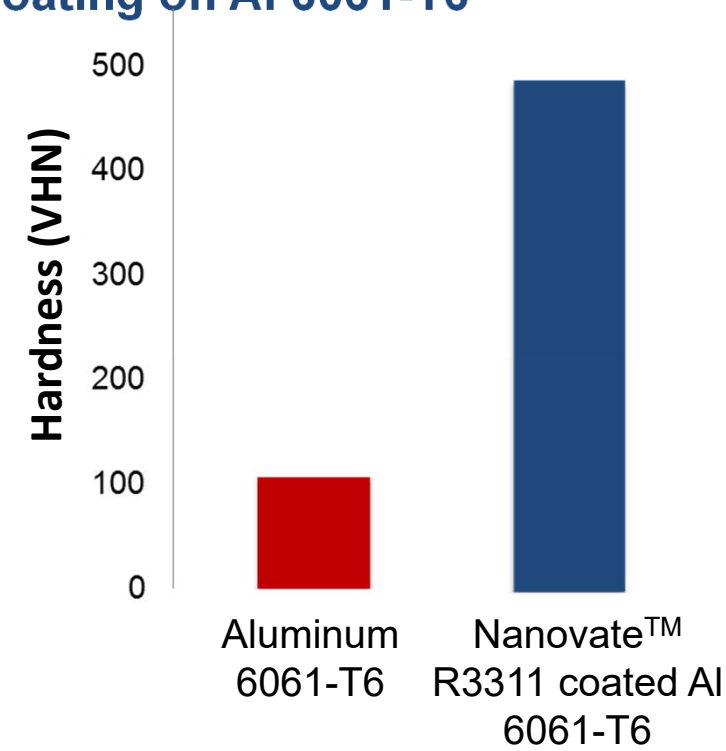
Plating conditions	Hardness (VHN)	Taber Wear Index (mg/1000 cycles, CS-17 wheels)	Pin on Disk (Al_2O_3 pin)	
			Average Coefficient of friction (μ)	Sliding wear rate* (10E-6 mm^3/Nm)
Standard Operating Conditions	528	18.3	0.58	12.4
Re-optimized Operating Conditions	540 - 553	18.0 - 18.5	0.49 - 0.63	12.8 - 13.7

Objective 1: Plating on Aluminum

- ***Objective**:* Enhance strength, maintain weight advantages
- ***Status***: CoP chemistry (R3311) developed to improve plating chemistry compatibility with Aluminum substrates

Flexural stress-strain curve and corresponding hardness graph demonstrating the added advantage of **50µm Nanovate™ coating on Al 6061-T6**





Al specimen cross-sectional dimensions: 5 cm x 0.75 cm

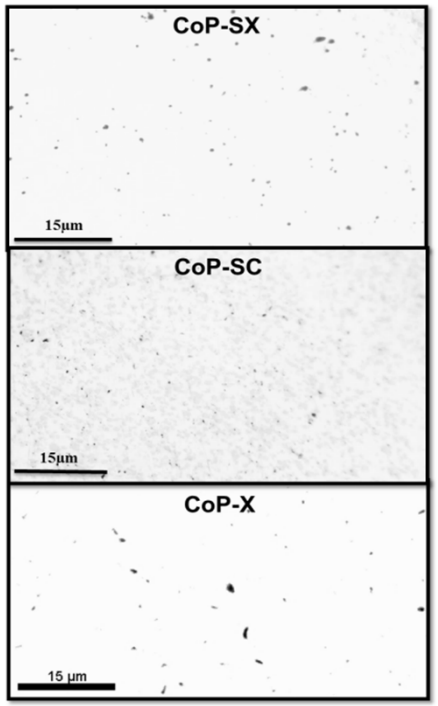
Objective 1: CoP Process Optimization

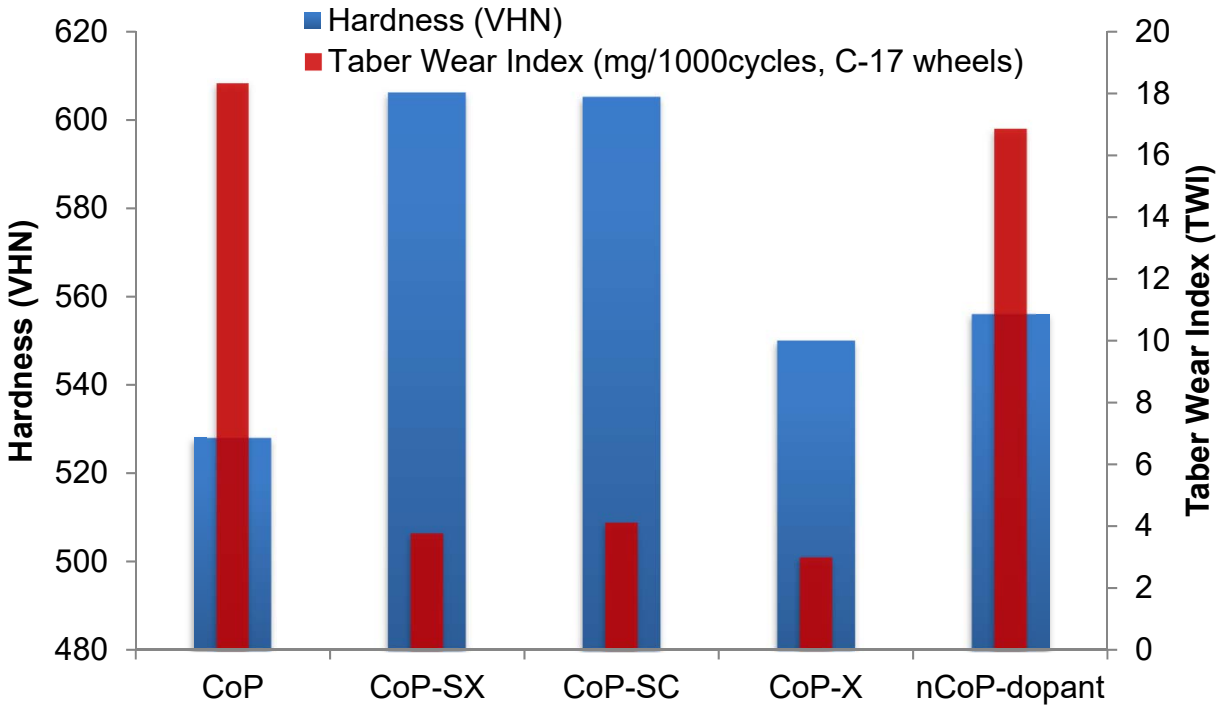
- CoP galvanic compatibility with Steel and Aluminum
  - CoP corrosion potential (OCP) close to steel and aluminum → reduced driving force for galvanic corrosion

Material/Coating	Open Circuit Potential
Aluminum Alloys	-800mV to -1000mV
4130 Steel	-680mV to -780mV
Nano CoP	-450mV to -550mV
Nickel 200	-100mV to -200mV

Objective 2: Property Enhancement via Nanocomposite Generation

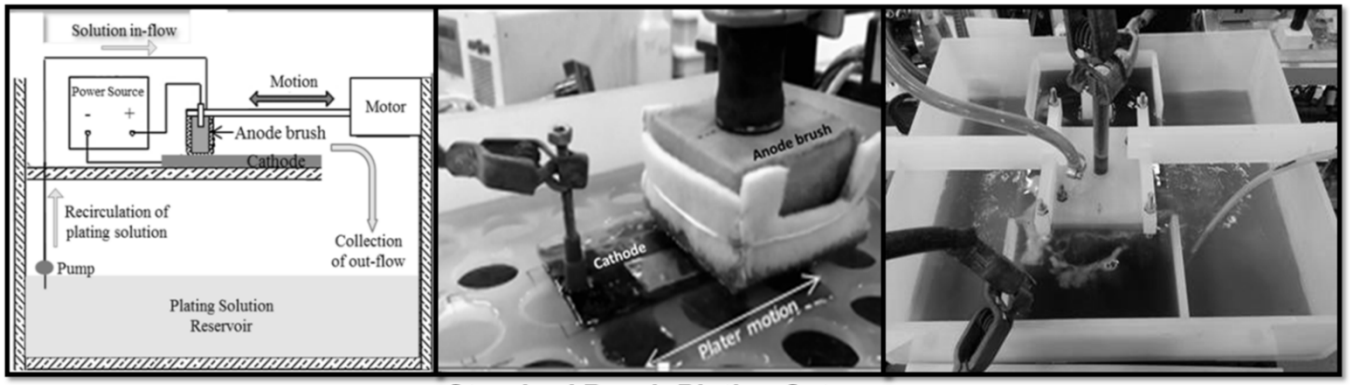
- Objective: Property enhancement via nanocomposite generation
- Status: Developed novel nanocomposite coating systems via co-deposition of suspended particles
  - Cirrus is developing novel in-situ particle formation and deposition of nanoparticles





Objective 3: Brush Plating Systems for Repair Operations

- Objective: Evaluate brush plating systems for repair operations using optimized materials
- Status: Successful deposition of CoP, co-deposition of suspended particles
  - Continuing development of specialized repair systems underway to improve flow systems and therefore deposit uniformity and properties



Standard Brush Plating Set-up

Property →	Hardness (VHN)		Taber Wear (mg/1000 cycles, CS-17 wheels)	
Set-up → System ↓	Conventional Plating	Brush plating	Conventional Plating	Brush Plating
CoP	528	513	18.3	18.9
CoP-SX	606	597	3.8	4.2
CoP-SC	605	582	4.1	5.7

Benefits to DoD / Summary

- Objective 1: CoP process optimization
  - DC-compatibility: reduced infrastructure costs associated with pulse plating
  - Lower operating temperature: reduced processing cost, improved ease of operation and compatibility with various masking waxes
  - Plating on aluminum: enhanced strength of aluminum with minimal weight addition
  - The R3010 and R3311 plating processes are suitable for plating various substrates including various steels, aluminum alloys, polymers and polymer composites

Benefits to DoD / Summary

- Objective 2: Nanocomposite generation
  - Improved deposit property: hardness and abrasive (Taber) wear performance
  - Feasibly translate to improved in-field performance and extended service life of component
- Objective 3: Brush plating systems
  - Selective repair of components with properties equivalent achieved via conventional tank plating
  - Provides process for possible field repair of components

Next Steps / Work in Progress

- Process stand-up (60L) for characterization, evaluation and validation of optimized
- Validation tests
  - Composition
  - Microstructure
  - Adhesion
  - Micro-hardness
  - Ductility
  - Wear
  - Corrosion
  - Hydrogen embrittlement
- Computational design and modelling of electrochemical deposition processes and corrosion prediction
  - Reduce risk in transitioning new electrochemical-based technologies

For additional information, please visit
www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Surface-Engineering-and-Structural-Materials/Composites-Alloys-and-Ceramics/WP-2609

Speaker Contact Information

McCrea@integran.com; 416-675-6266 (ext. 235)

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Q&A Session 1

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Qualification Testing of Electroformed Nanostructured Cobalt-Alloy Bushings

Dr. Alan Grieve
U.S. Naval Air Systems Command

SERDP DOD • EPA • DOE | ESTCP

Agenda

- Project background
- Overview of nanostructured materials
- Technical approach
- Progress to date
- Conclusions

Project Background

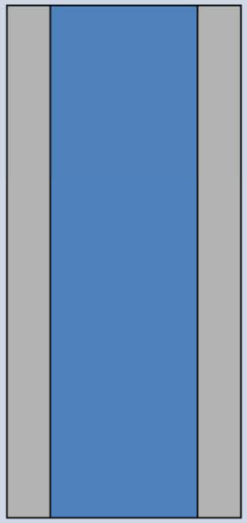
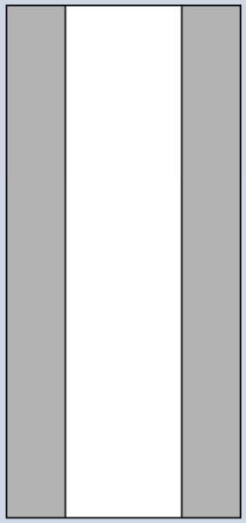
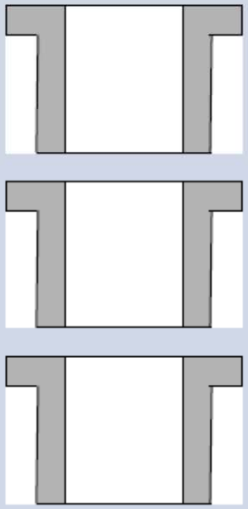
- Copper-beryllium (Cu-Be) is a commonly used high-strength bushing material
- High strength, good wear properties
- Machining creates Be-containing dusts and fumes
- Be dusts are toxic and can lead to lung cancer and chronic beryllium disease (CBD)
- Less toxic alternatives are needed

Nanostructured Alloys

- Nanovate cobalt-phosphorus (CoP) alloy
  - Favorable properties as bushing materials
  - Fabricated using an electroforming process
  - Near net-shape fabrication
  - Suitable for large and small diameter bushings

Electroformed Nanometal Bushings

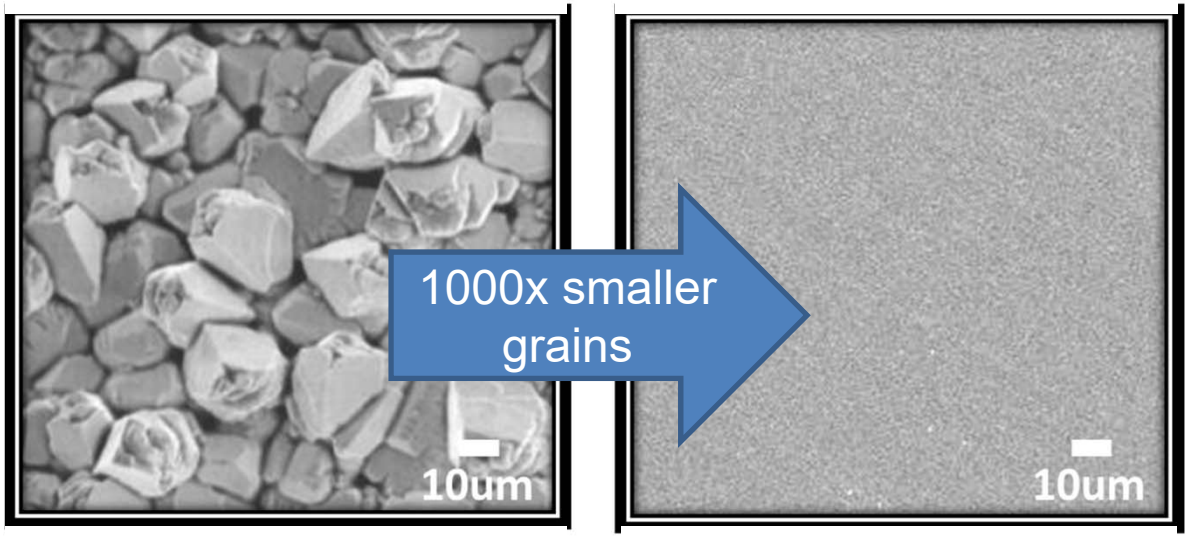
- Near net shape manufacturing process
- Cost effective: Less material waste during machining

Step 1 Electroform desired thickness (~0.25”) onto temporary mandrel	**Step 2** Remove mandrel	**Step 3** Machine bushings
		

Advantages of Nanostructured Alloys

- Hall-Petch relation
  - Decreased grain size, increased strength
  - Grain boundaries act as pinning points impeding propagation of dislocations

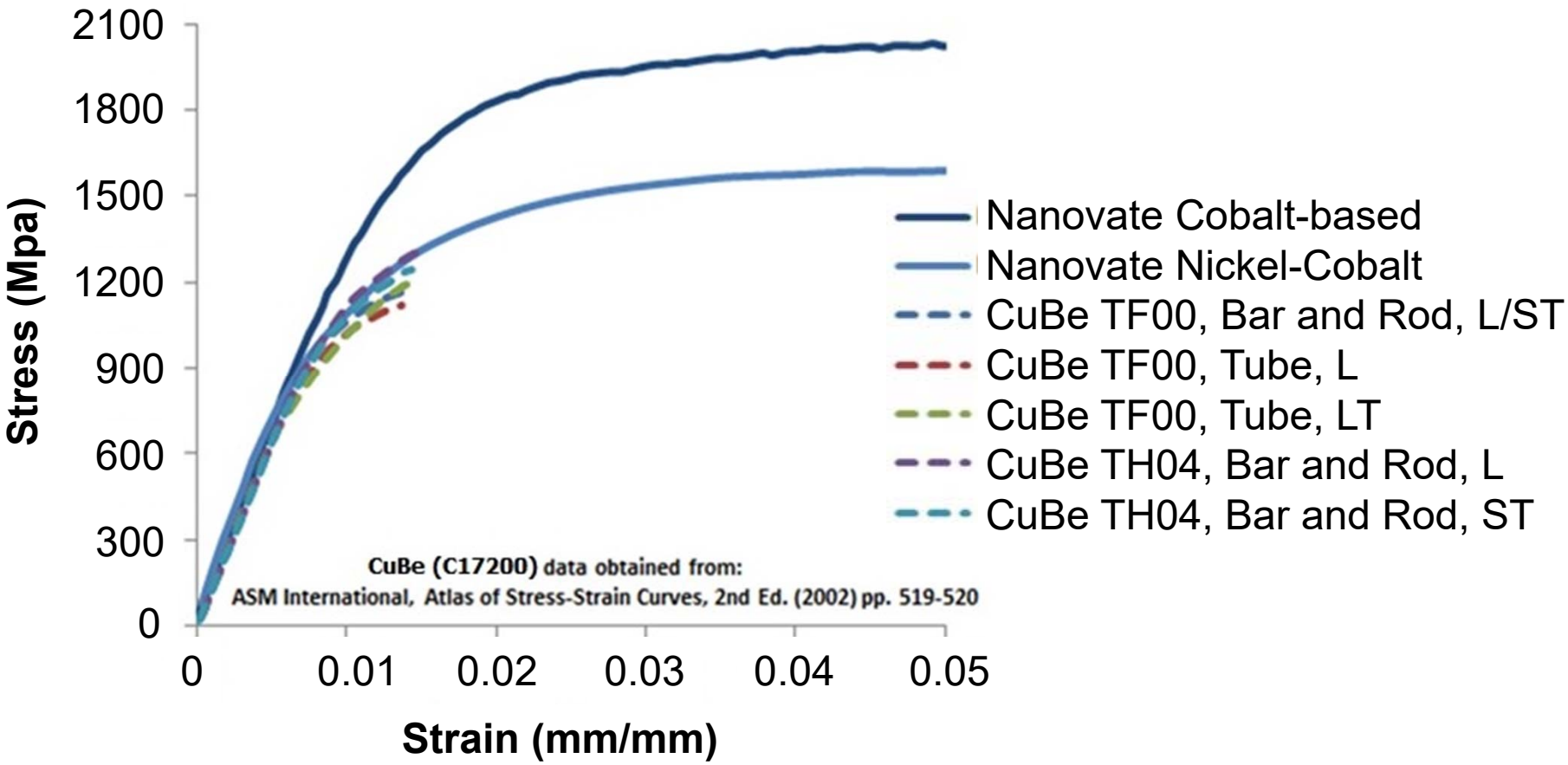
Conventional metals | **Nanovate™ metals**



Grain size = 10-100 μm | Grain size ≦ 20 nm

Uniaxial Tensile Test Data

- Tensile performance of Nanovate cobalt- and nickel-cobalt alloys is superior to copper-beryllium

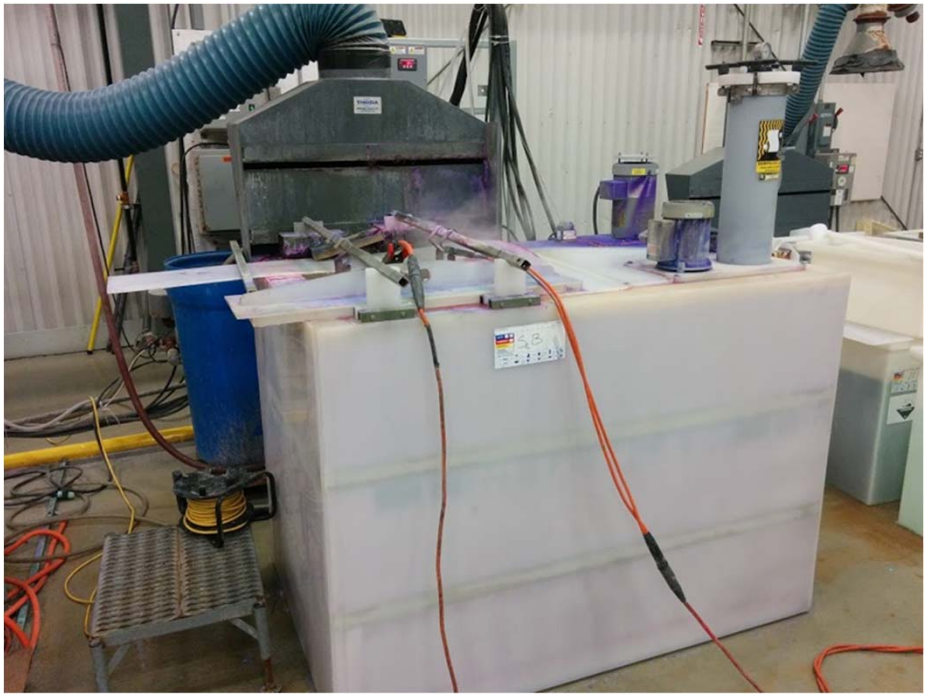


Technical Approach

- Integran and NAVAIR are primary performers
- Five major tasks
  1. Manufacturing process definition / demonstration
  2. Property and performance testing
  3. Depot evaluation and insertion
  4. Cost and environmental analysis
  5. Material qualification
- Results from the first two tasks will be discussed here

Tank Installation at Integran

- Design, construct, and install a dedicated bushing fabrication tank (volume ~1,500 L)
- Specifications
  - 5 ft x 3 ft x 4 ft
  - Double-wall insulation
  - Auto-level water fill
  - Out-of-tank filtration
  - Out-of-tank circulation



Optimize Electroform Process

- Goal was to optimize process for electroforming of nano-CoP bushings
- Design reusable electroform mandrel
- Optimize key process parameters
  - pH
  - Current density
  - Rotational speed

Mandrel Design and Optimization

- Four different designs investigated
  - Metal sleeve with collapsible interior core
  - Metal sleeve with polymer core
  - Unsupported, thin stainless steel mandrel
  - Metallized polymer core

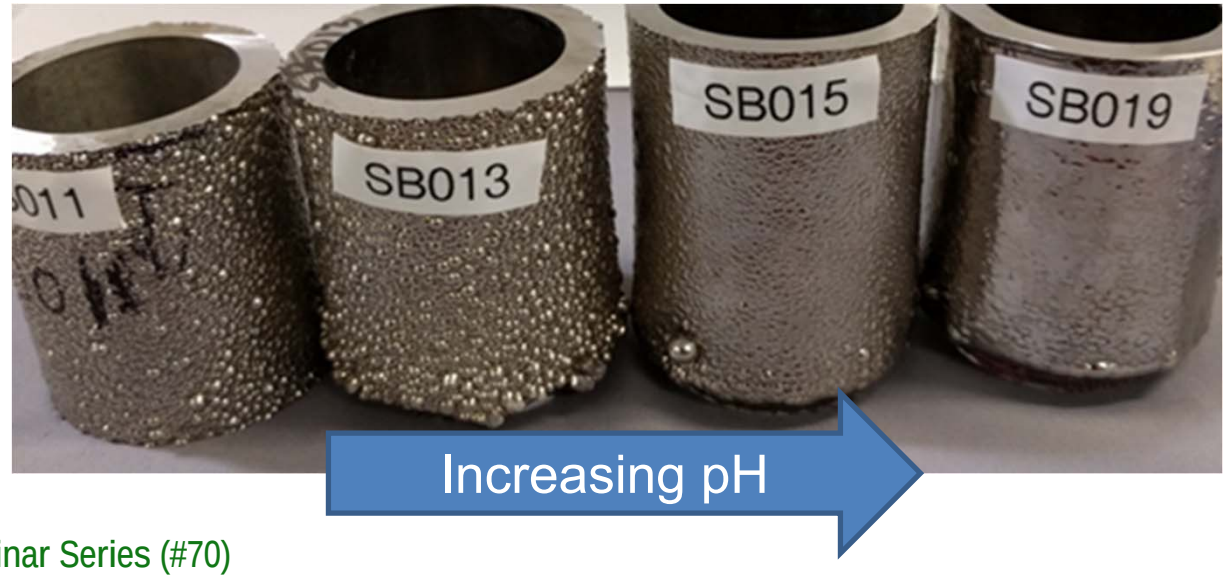
Mandrel Design and Optimization

- Selected criteria were used to assess each design along with a ranking for each

Criteria	Design 1	Design 2	Design 3	Design 4
Reuse	Partially	Partially	No	Yes
Durability	High	Low	High	Medium
Ease-of-use	High	Low	High	High
Surface quality	Good	Good	Good	Good
Cost	Medium	Medium	High	Low

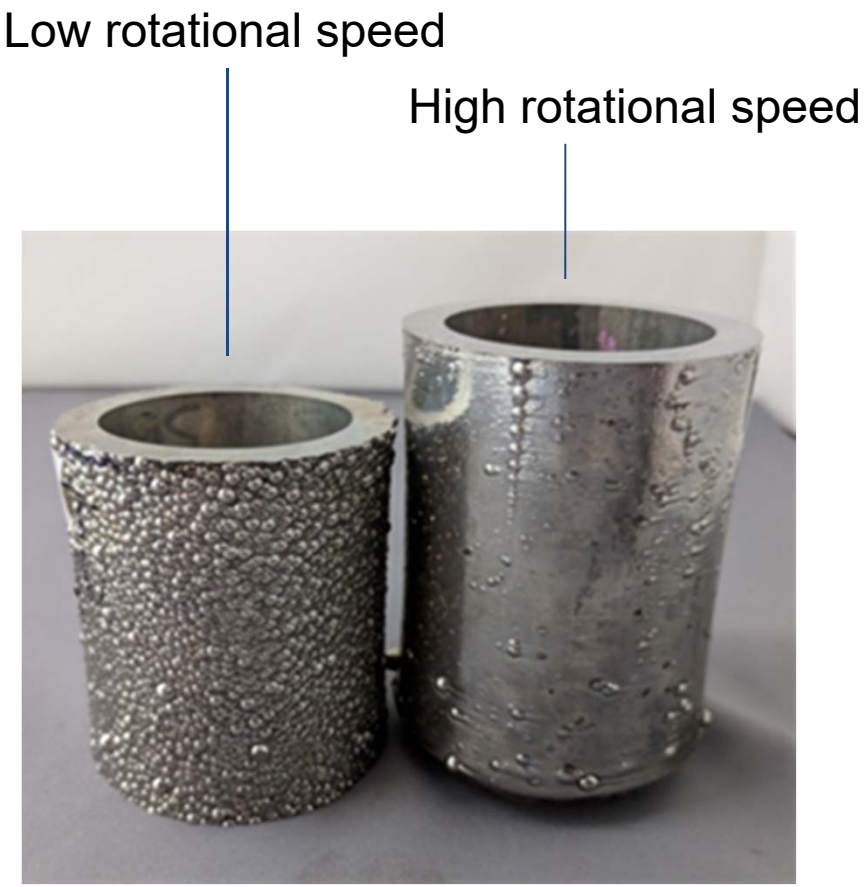
Process Optimization

- pH
  - At low thickness, surface roughness not affected by pH
  - At higher thickness, increase in pH decreases surface roughness



Process Optimization

- Rotation speed
  - Low thickness
    - Surface roughness not affected by speed
  - Higher thickness
    - Increased rotational speed decreases surface roughness

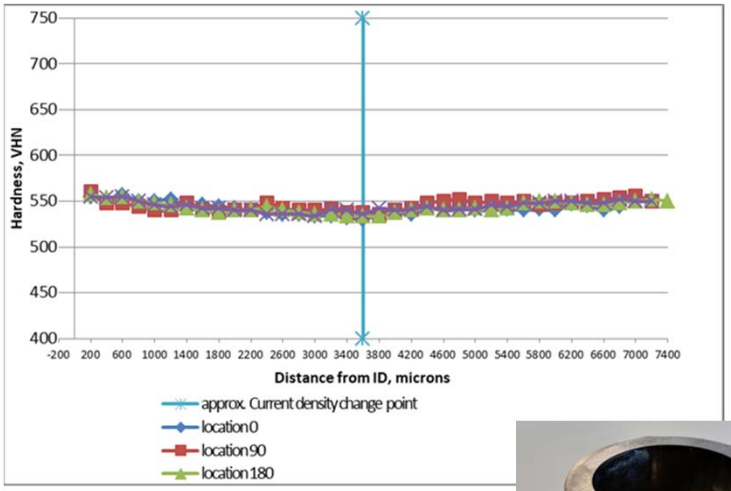


Process Optimization

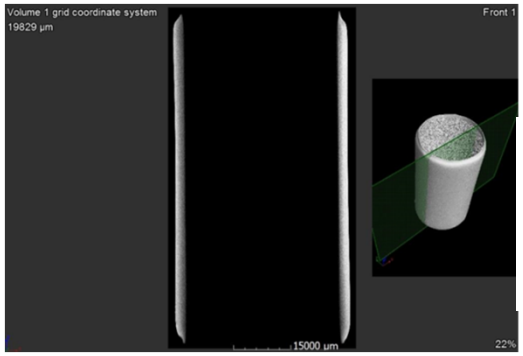
- Current density
  - High current densities are used in electroforming
  - Surface roughness observed with increased build-up
  - Adjustment of the current density during the process eliminates this problem
- Primary process parameters now optimized

Process Verification

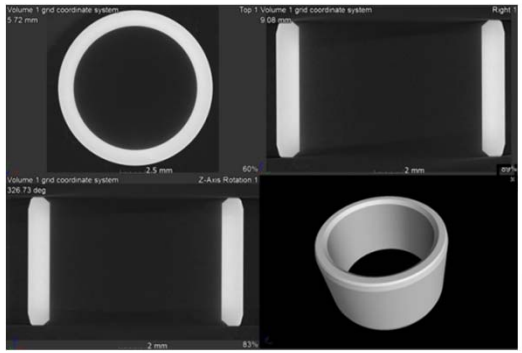
- Hardness
  - Uniform hardness through the deposit



- NDI
  - CAT scan inspections found no voids



As-fabricated "blank" material



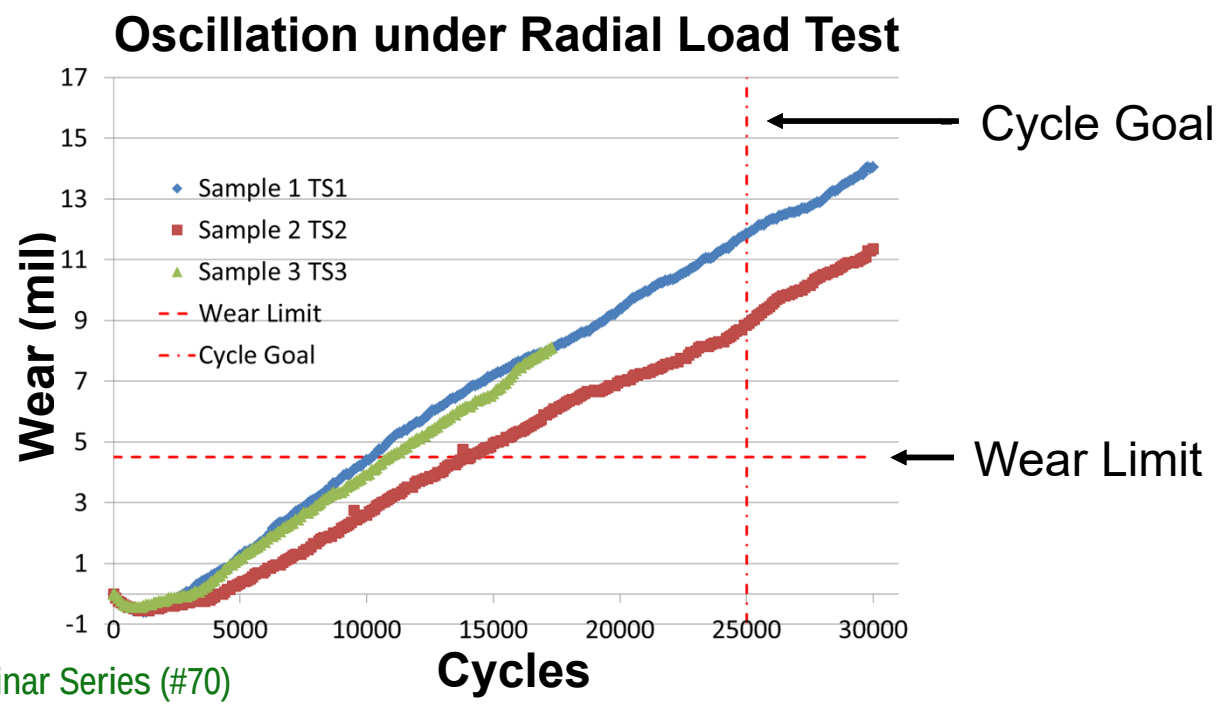
Finished, machined bushing

Property and Performance Testing

- Developed joint test protocol
  - Mechanical testing
    - Tensile, compression, fatigue
  - Subscale bushing testing
  - Galvanic compatibility testing

Subscale Bushing Testing

- Preliminary subscale bushing tests performed
  - Tested according to SAE AS 81934 Rev.B
  - Normally applied to Teflon-lined bushing

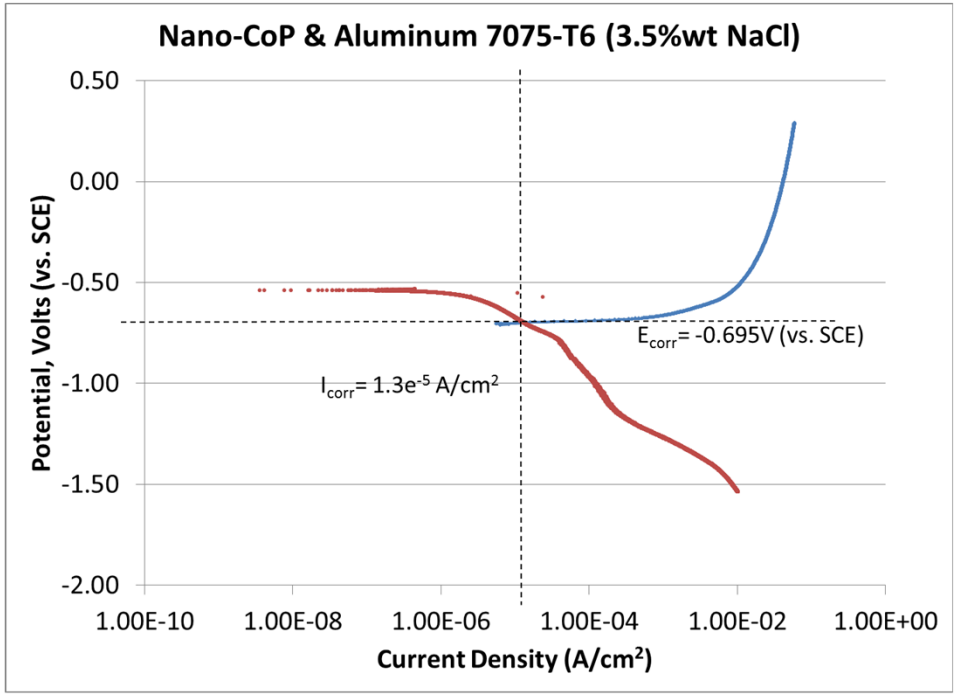


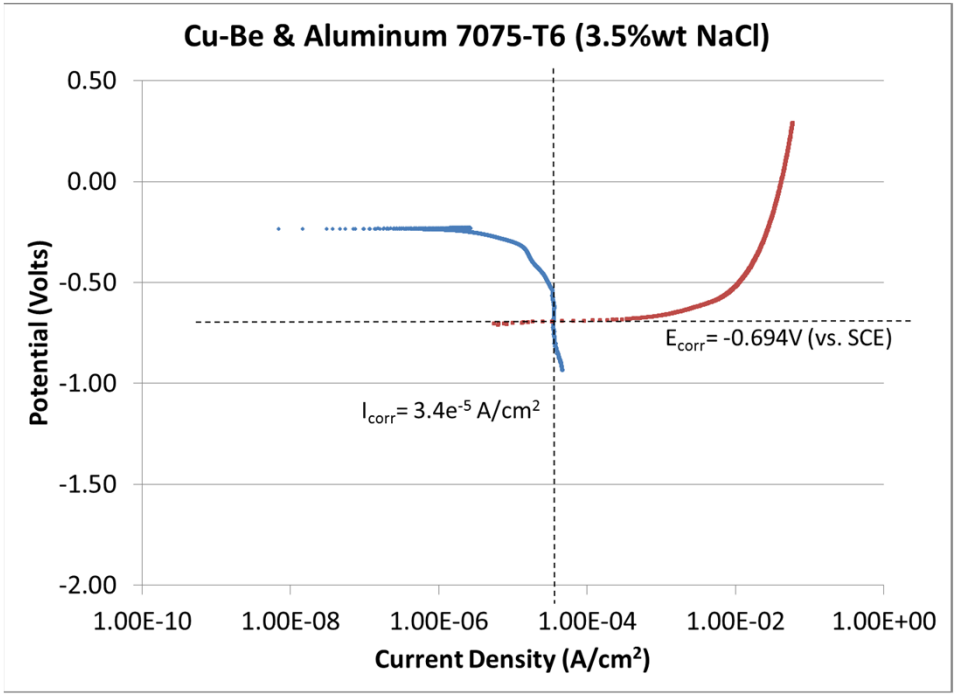
Subscale Bushing Testing

- Absence of liner meant wear requirement was not met
  - Bushing showed linear wear, with little distress on wear surface
  - Low sound generation for metallic bushings

Galvanic Compatibility Testing

- Polarization studies of n-CoP, CuBe and Al7075 indicate improvements in galvanic compatibility between n-CoP and Al alloys
- Lower corrosion current with n-CoP/7075 than CuBe/7075
- Similar improvements seen with stainless steel alloys





Depot Evaluation and Insertion

- Goal is to transition technology to FRCs
  - Initially at FRC-SE (Jacksonville)
- Stand-up electroforming tank
- Perform machining evaluations
- Bushings on the arresting tail hook of the Naval trainer identified as component for demonstration/validation
- Looking at ground vehicle applications

Potential DoD Benefits

- Potential to replace toxic Be-containing bushings in high-load applications
  - Less hazards for DoD depot personnel
- Potential to remove the need for Be-machining operations from DoD depots
  - Reduce ESOH related costs
- On-demand, near net shape bushing fabrication capability

Conclusions

- An optimized electroforming process for fabrication of aircraft bushings was demonstrated
- Materials are void-free and have uniform hardness
- Initial bushing tests indicated promising properties
- Potentially more favorable galvanic properties
- Test protocol for complete evaluation developed

For additional information, please visit

www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Surface-Engineering-and-Structural-Materials/Composites-Alloys-and-Ceramics/WP-201624

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Q&A Session 2

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The next webinar is on April 19, 2018

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Survey Reminder

Please take a moment to complete the survey that will pop up on your screen when the webinar ends

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